Multilens Contact Adhesion Tests

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University of Massachusetts - Amherst

NCMC – Adhesion Workshop

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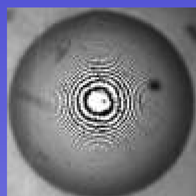
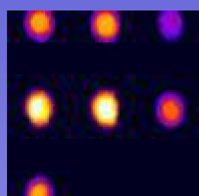
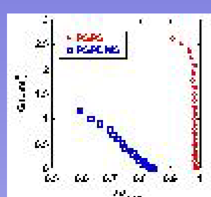
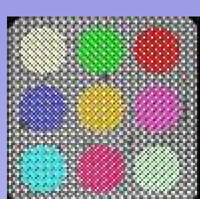
Outline









- Conventional Adhesion Tests
- Multilens Contact Adhesion Test Theory and Method
- Examples and Implementation
- High-Throughput Analysis
- Practical Points
- Summary

Understanding Polymer Adhesion

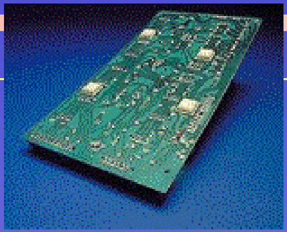
Motivation

- "Bolts and screws can be modeled with software... , but glue makers have yet to come up with a predictive model", **Forbes, 10.29.01**

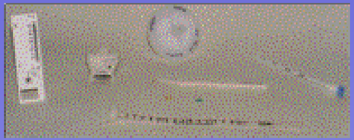
- Myriad of variables control adhesion

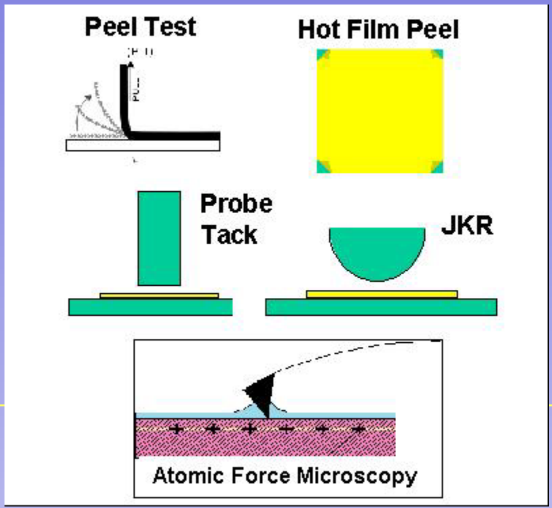
- Existing techniques


Aerospace


Electronic Packaging


Automotive


Biomedical



- Surface Energy
- Molecular Weight
- Time
- Temperature
- Humidity
- Roughness
- Geometry

Peel Tests

Advantages
- Simulates typical use
- Easy sample preparation
- Semi-quantitative results
- Customer-friendly results

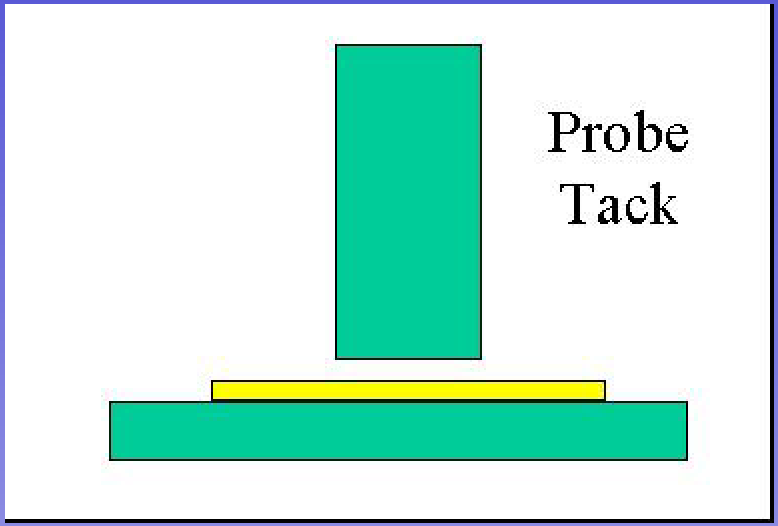
Disadvantages
- Results are not absolute
- Sample preparation difficult to standardize
- High statistical populations required
- Stress distribution complicates analysis (backing, adhesive, peel front)

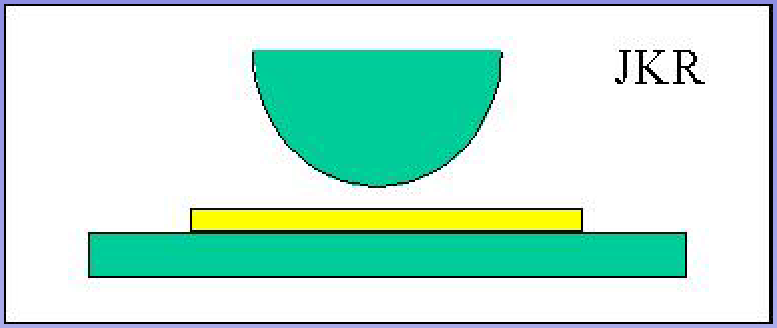
Image from www.quadgroupinc.com

Axisymmetric Adhesion Tests

Advantages
- Removes backing influence
- Standardizes sample prep
- Bulk and interfacial contributions can be decoupled

Disadvantages
- Absolute analysis is time-consuming
- Consumer knowledge is limited
- Backing properties must be measured separately





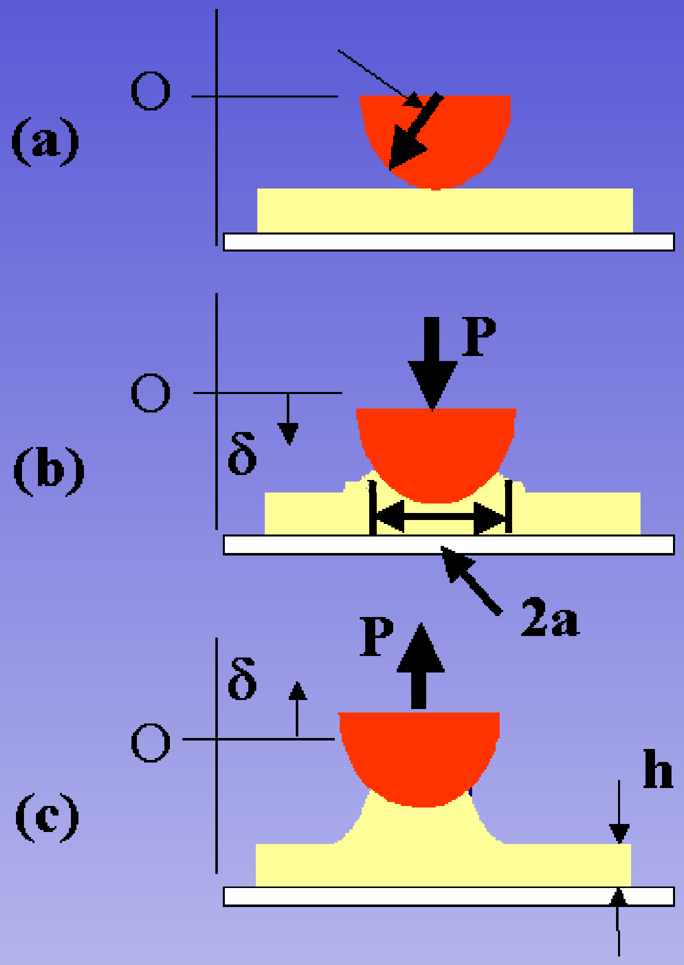
Typical Probe-type Adhesion Tests

General Procedure

1. Place adhesive on rigid substrate
2. Position probe above adhesive
3. Move probe into contact with adhesive
4. Hold in contact for arbitrary time
5. Separate probe from adhesive

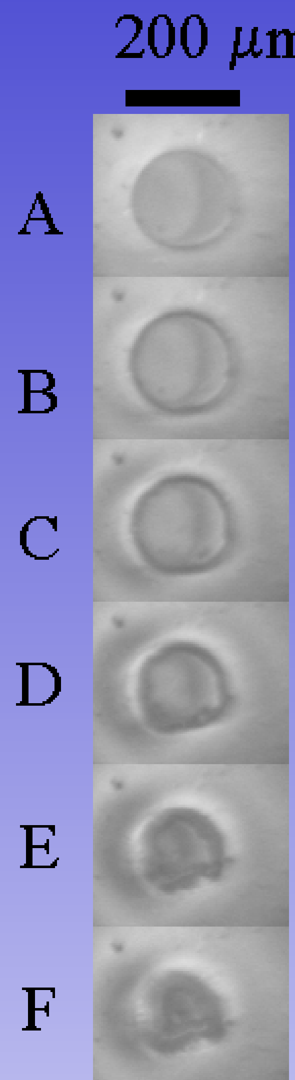
Typical Measurement Data:

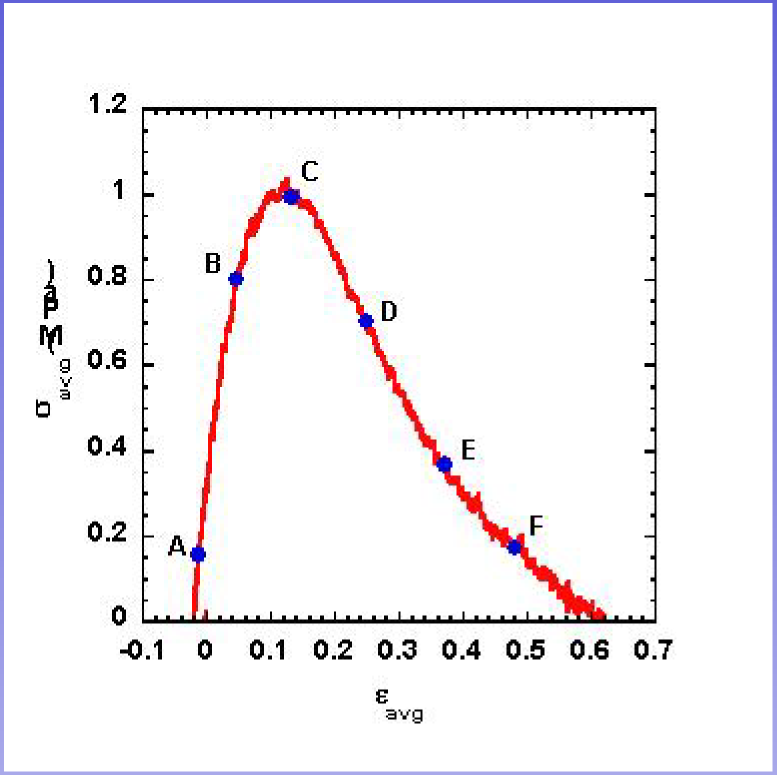
Applied Force
Displacement
(Contact Area)



Spherical Probe

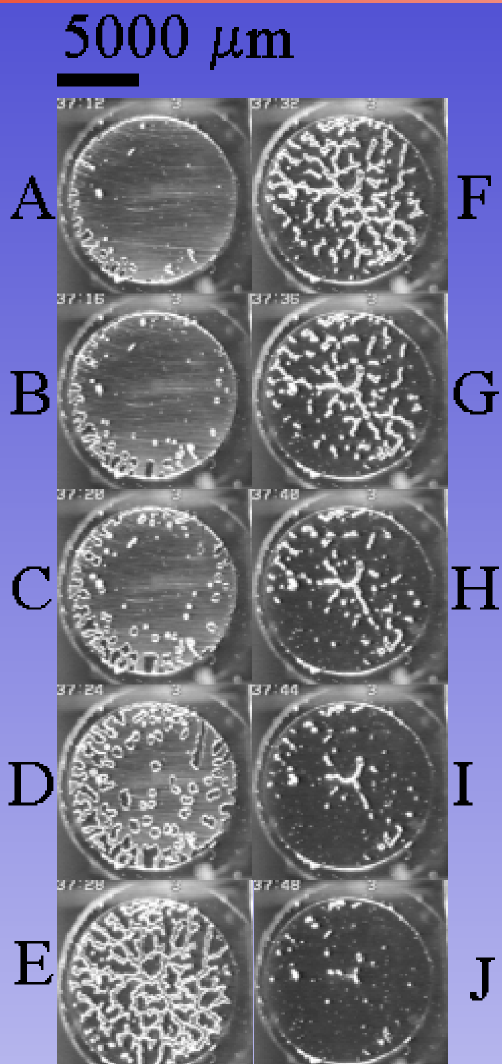
Low Confinement

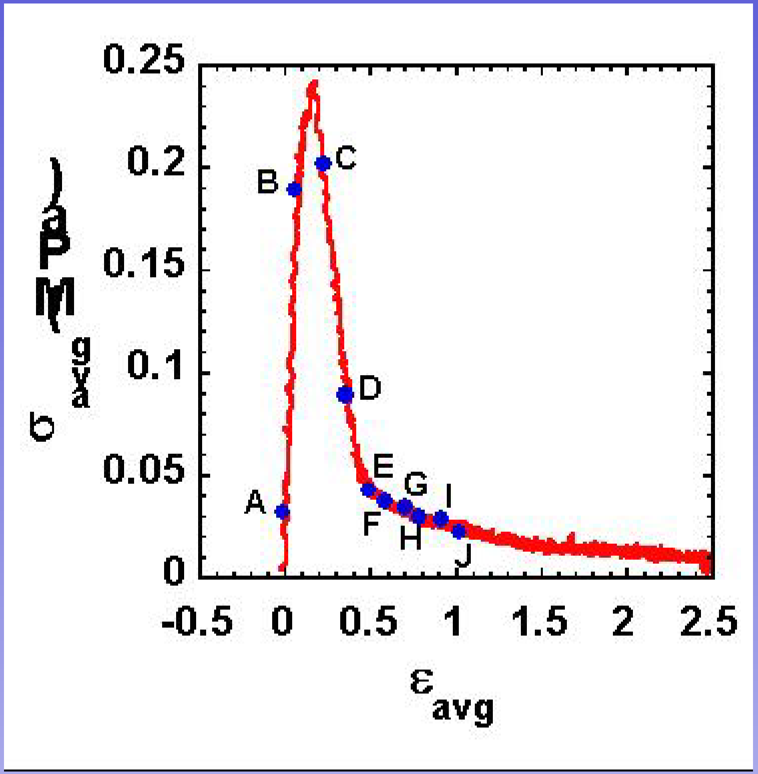




Flat Probe

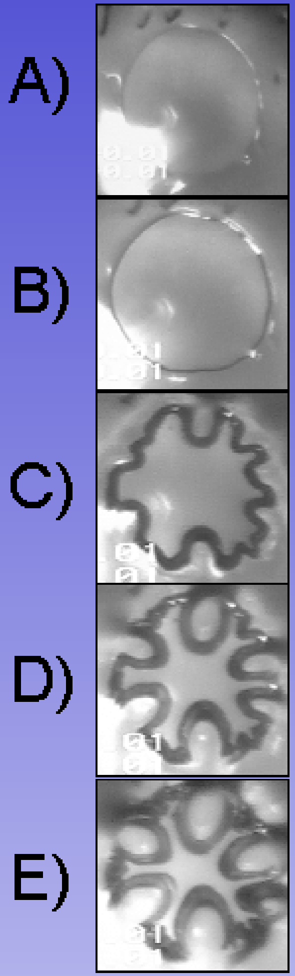
High Confinement

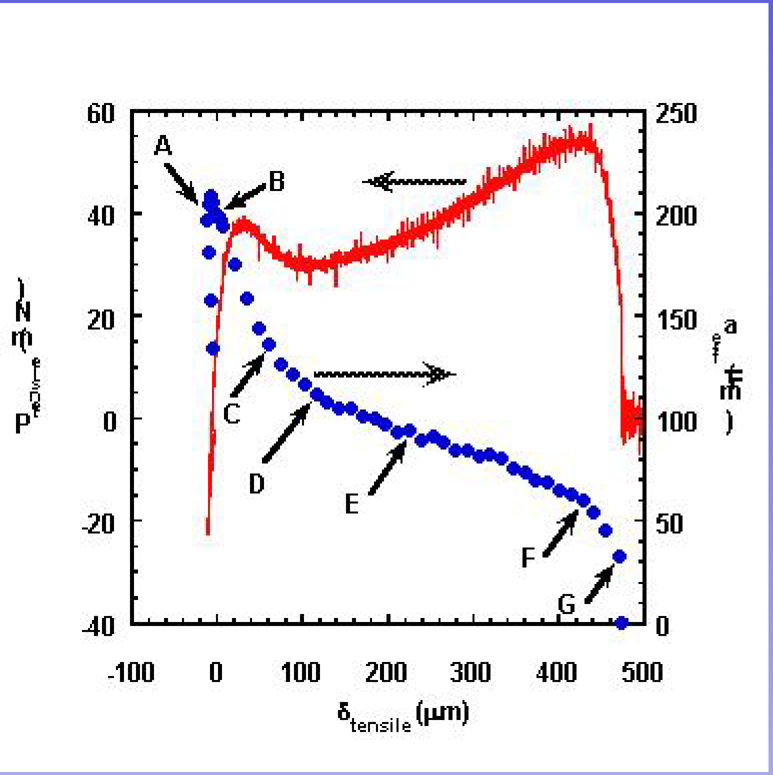




Spherical Probe

Medium Confinement





Analyzing Probe Tests

Total Dissipation, $W_{adh\backslash}$

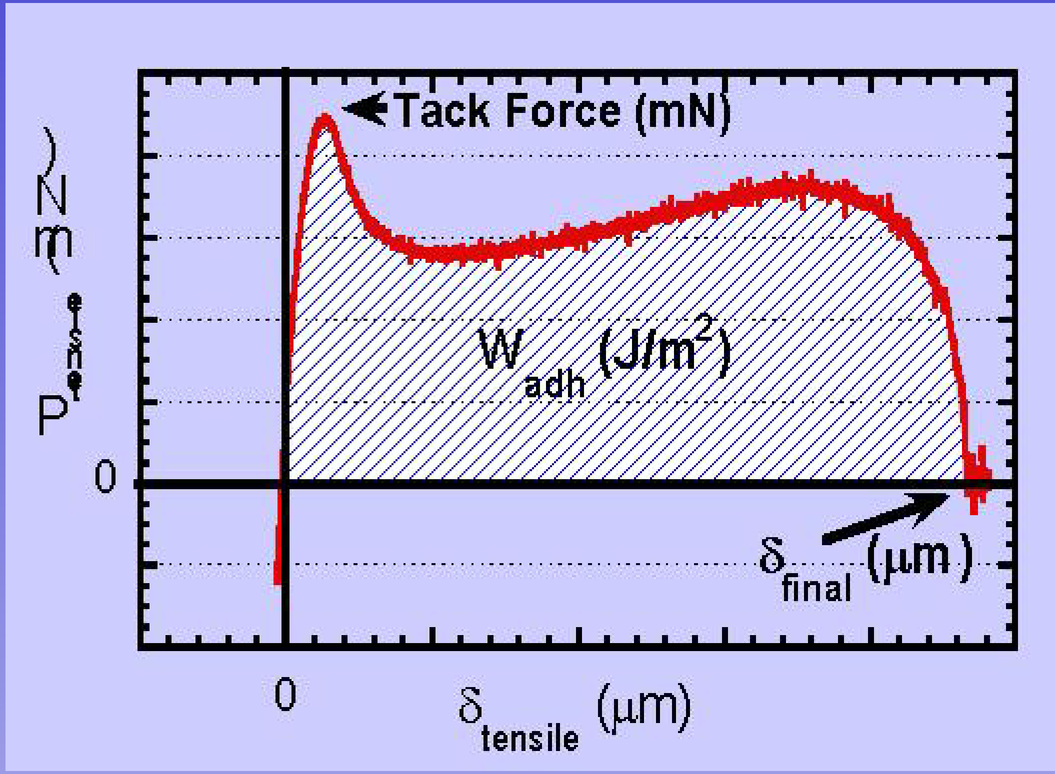
- Total area under curve normalized by contact
- Correlates with peel energy

Tack Force, P_{tack}

- Maximum tensile force
- Typically associated with instability initiation

Strain at Failure, δ_{fail}

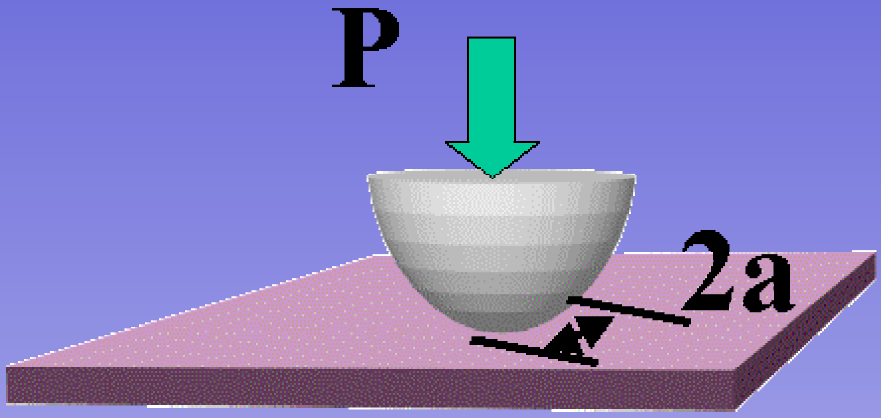
- Maximum tensile strain

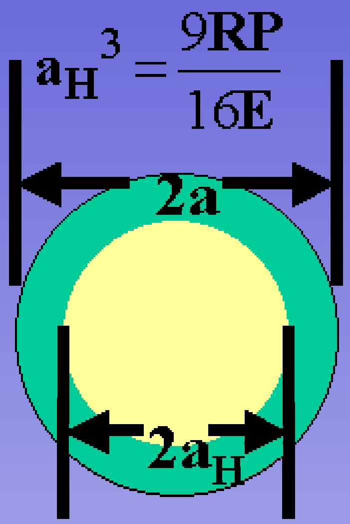


- Quantities are very useful for relative measurements
- Standardization is established, ASTM -
- Contact area images can provide great insight

Theory of Johnson, Kendall, and Roberts (JKR)

General Idea





JKR Equation ⟶

$$a^3 = \frac{9R}{16E}\left[P + 3\pi GR + \sqrt{6GRP + (3\pi GR)^2}\right]$$

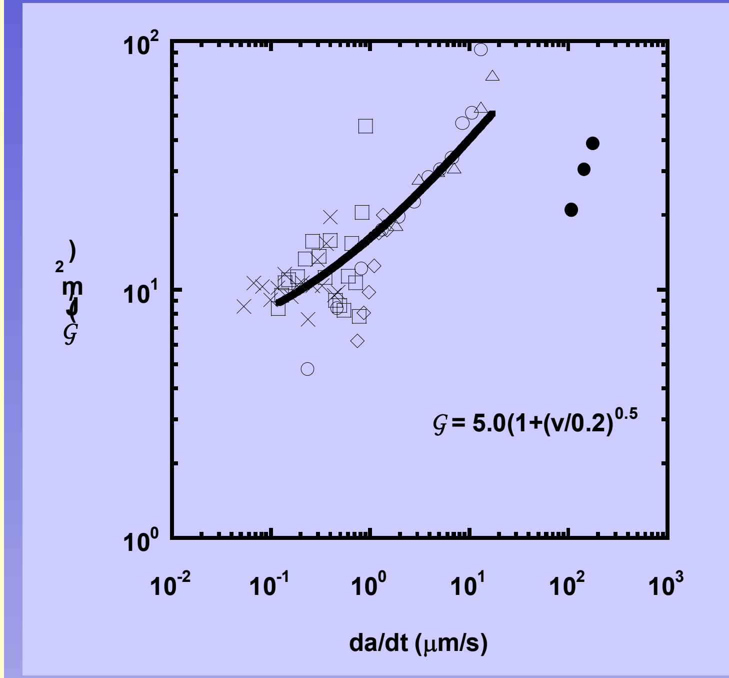
Details of Analysis

1. Measure radius of curvature, R
2. Record force, P
3. Measure contact radius, **a**
4. Correlate **a** & P data
5. Plot **a**^{3} vs. P and either:
   - Use two parameter fit to determine E and $\mathcal{G}$

   - or -
   - Measure E independently and determine $\mathcal{G}$

   - or -
   - Record δ in addition to independently determine E and $\mathcal{G}$

6. Plot G vs. d**a**/dt to define material property



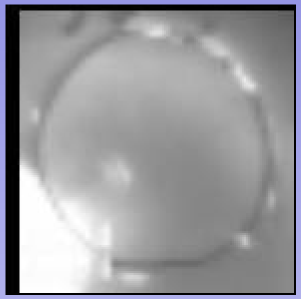
Special Cases

Finite-Size Corrections (for a>h)

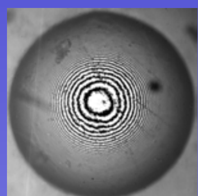
Shull, K.R., et al, *Macromol. Chem. Phys.*, 1998, **199**, 489-511.
Crosby, A.J. et al, *Journal of Applied Physics*, 2001, **88**, 2956-2966.

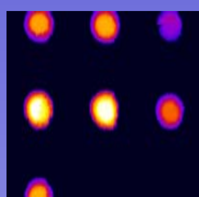
Viscoelasticity Corrections

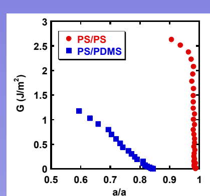
Lin, Y.Y., et al, *Journal of Applied Physics*, 1999, **32, 2250-2260.**
Johnson, K.L., ACS publication, 2000.

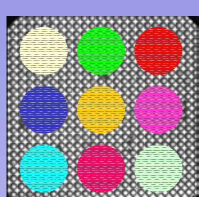



Outline









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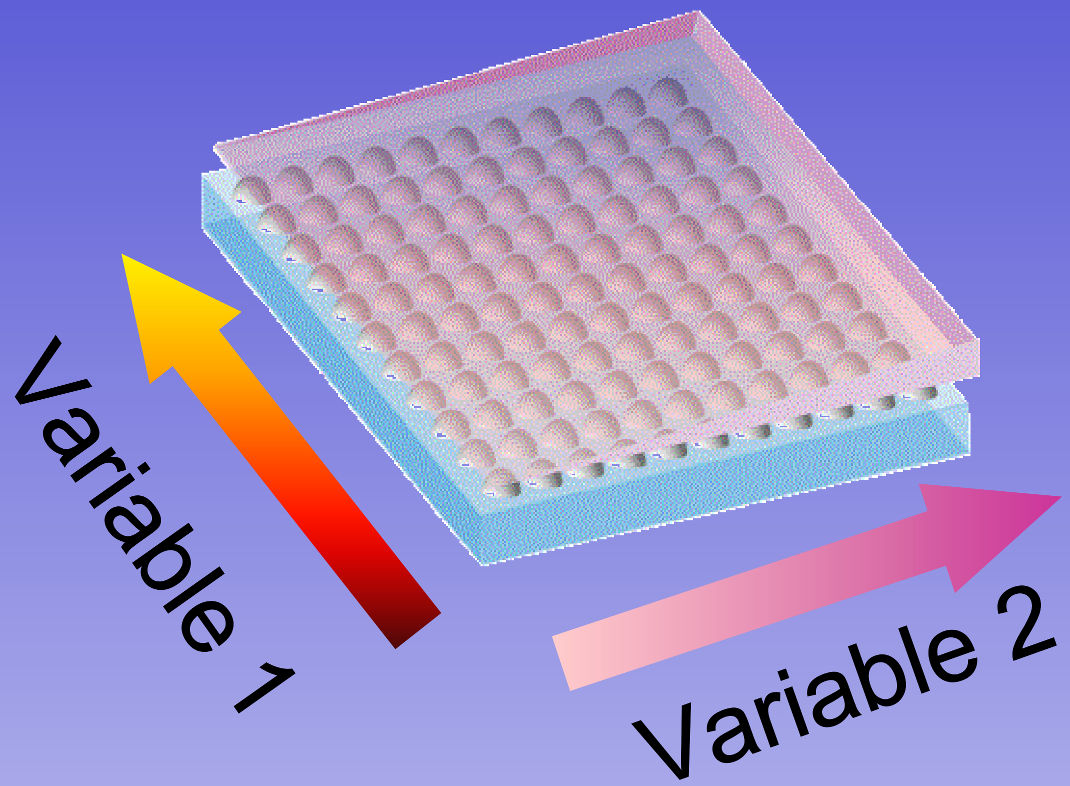
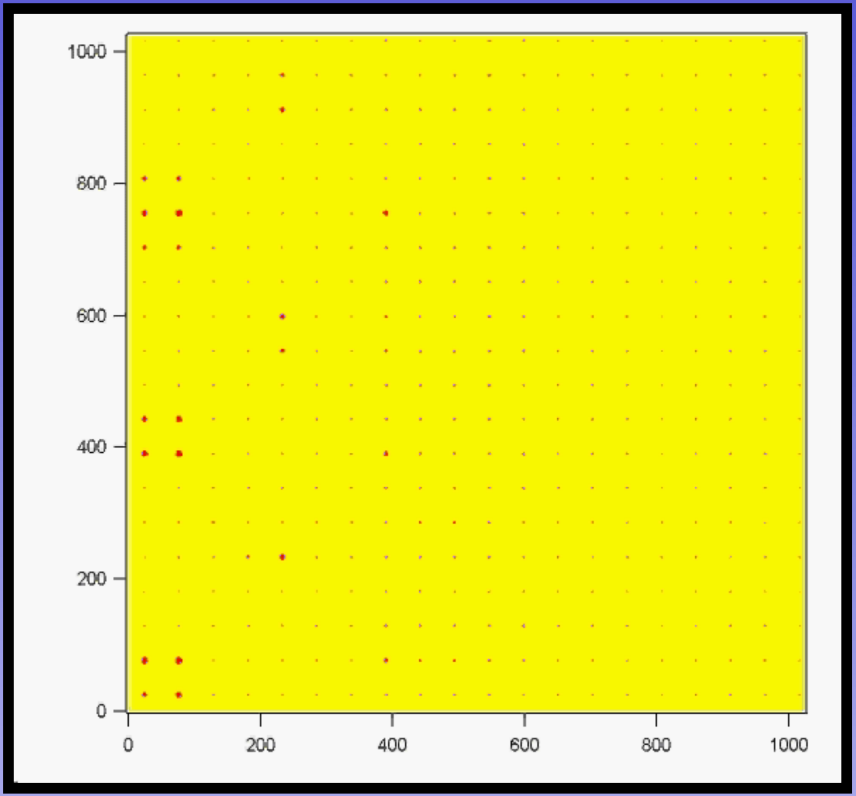
A *Combinatorial* Adhesion Test: MCAT

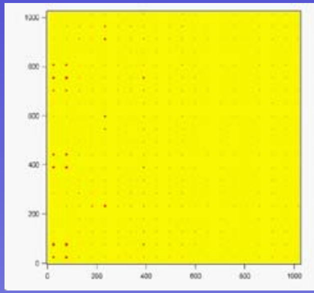


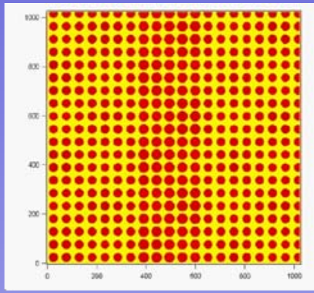
- **Measure a, d**
- **Determine G**

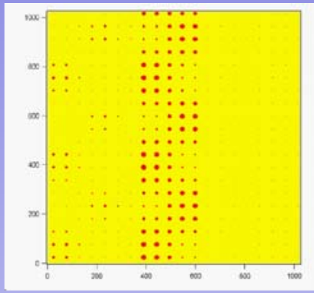
- **Possible Variables:**
  - **Temperature**
  - **Thickness**
  - **Strain**
  - **Surface Energy**

Qualitative Analysis









- Contact history images give qualitative information.

- Quickly map the relative adhesion of a surface or library.

How do we calculate $\mathcal{G}$?

$$G = \frac{3\left(P' - P\right)^2}{32\pi E \bullet a^3} \cdot f_P\left(a, h\right)$$

$$C = \frac{3}{8Ea} = \frac{d\delta}{dP} = \frac{\delta' - \delta}{P' - P}$$

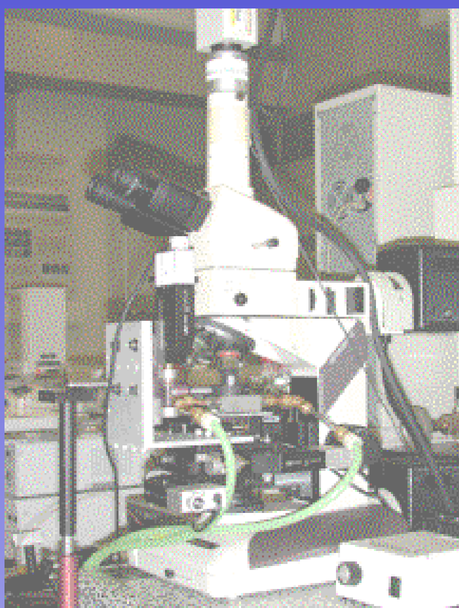
$$\boxed{G = \frac{2E\left(\delta' - \delta\right)^2}{3\pi a} \cdot f_\delta\left(a, h\right)}$$

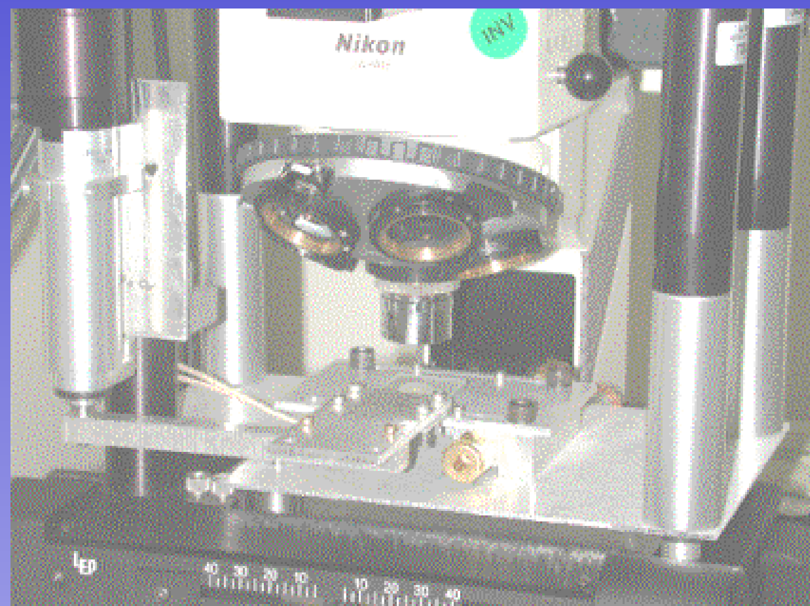
- Minor misalignment tolerable
- Each lens has a unique δ_0
- E must be measured independently or report $\mathcal{G}$/E
- Modify for confinement or viscoelasticity

*** K.R. Shull, et al., *Macro. Symp.*, 1997.

The Instrument

Time, cost, and precision

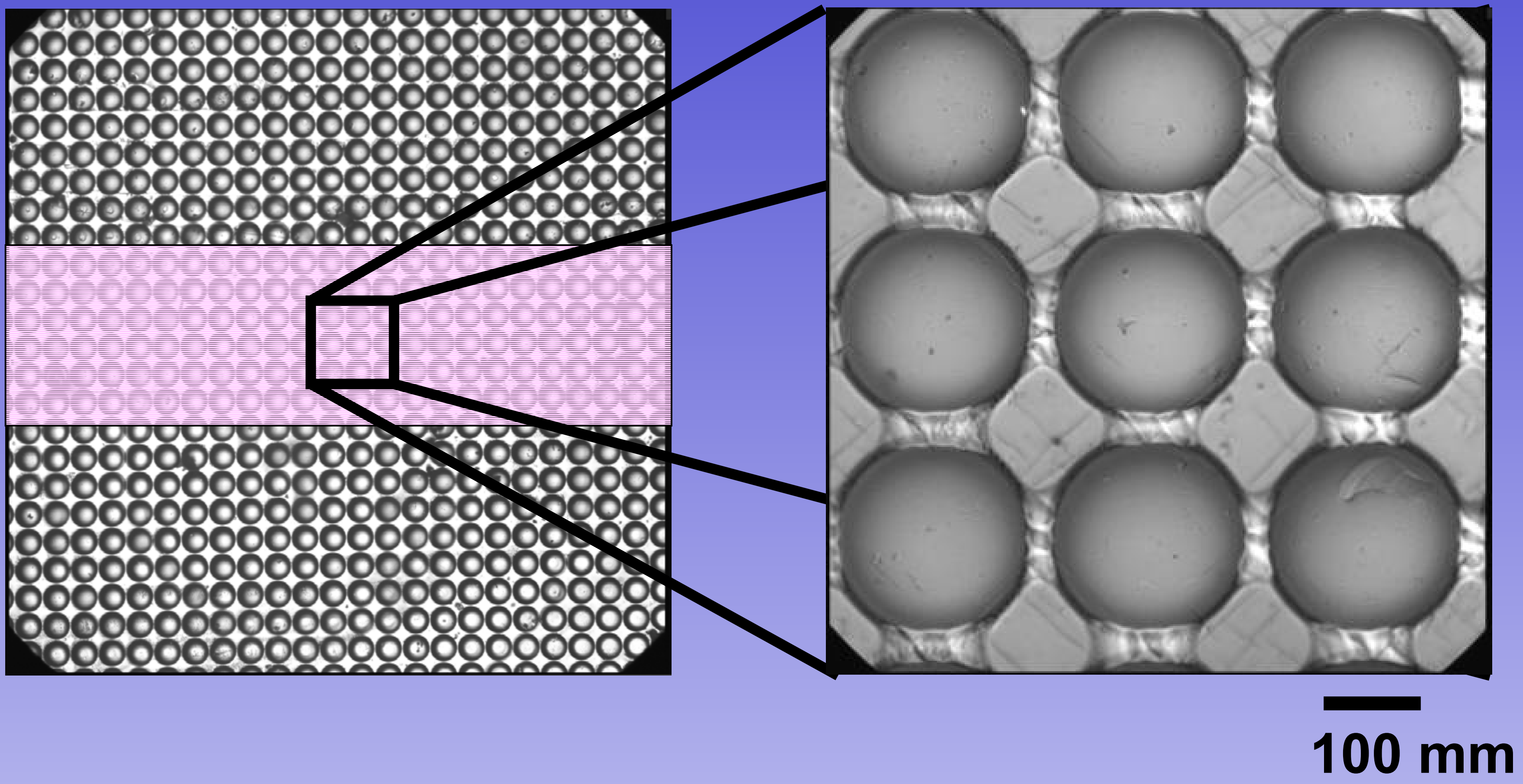




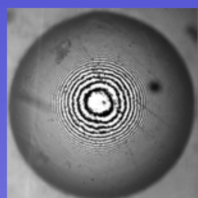
Main Components

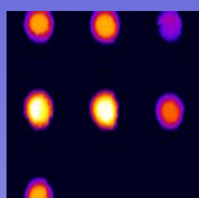
- Imaging System (I.e. microscope)
- Automated x-y stage
- Displacement control (i.e. actuator or micrometer)
- Displacement sensor
- Computer with DAQ card
- Alignment system (optional)
- Load cell (optional)

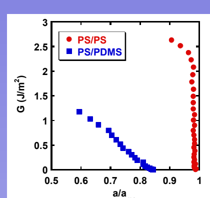
The Libraries

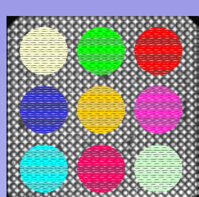


Outline









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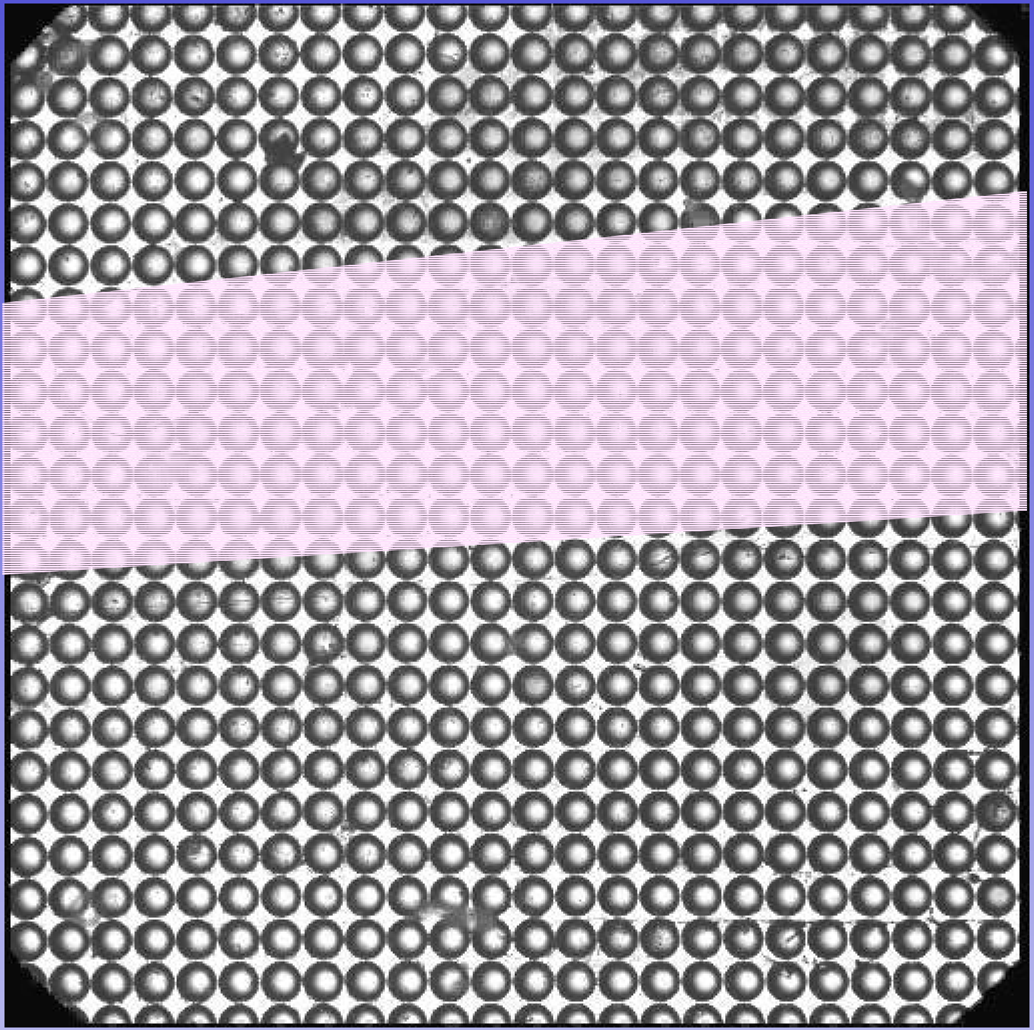
Contact Experiment



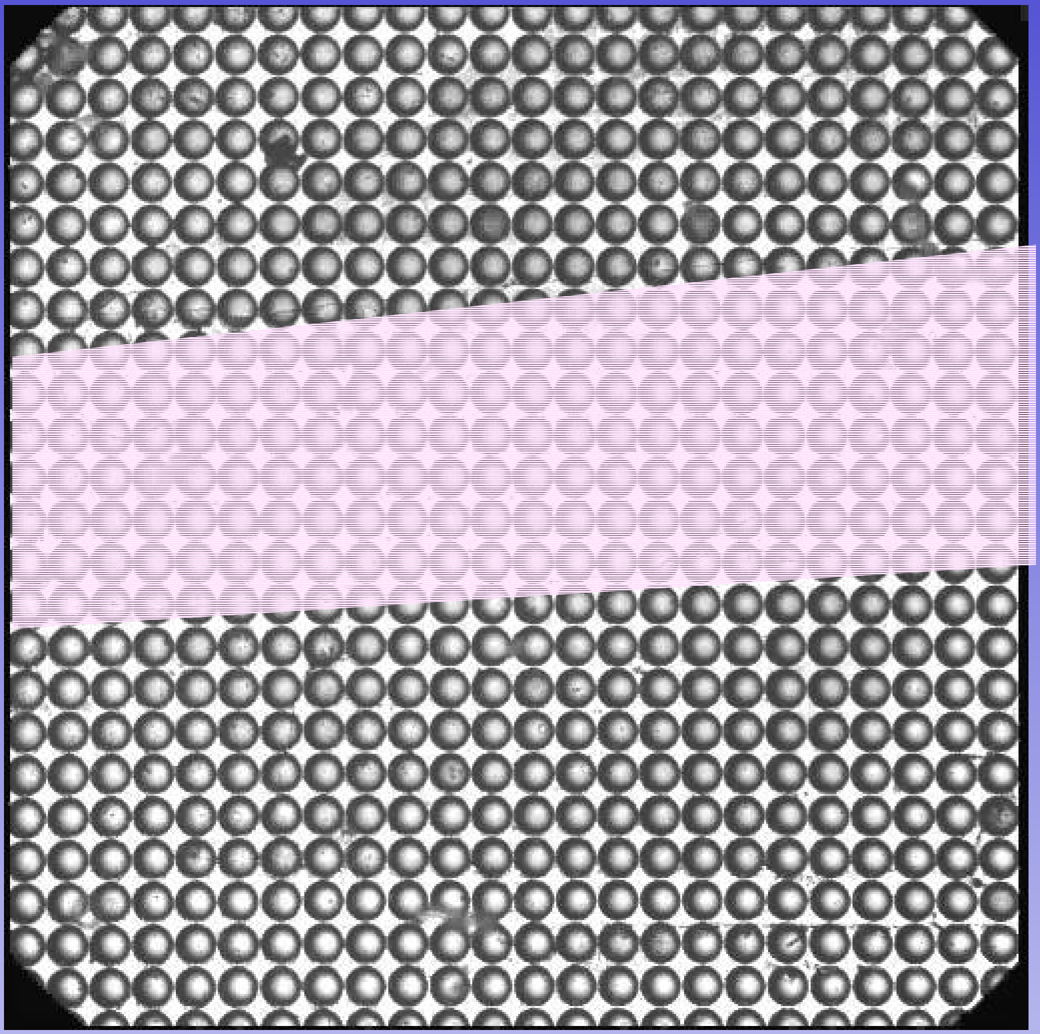
Conditions:

dd/dt = 1 mm/s

$h_{PS\ strip}$ = 30 nm

Temperature = 25°C

Contact at Elevated Temperatures



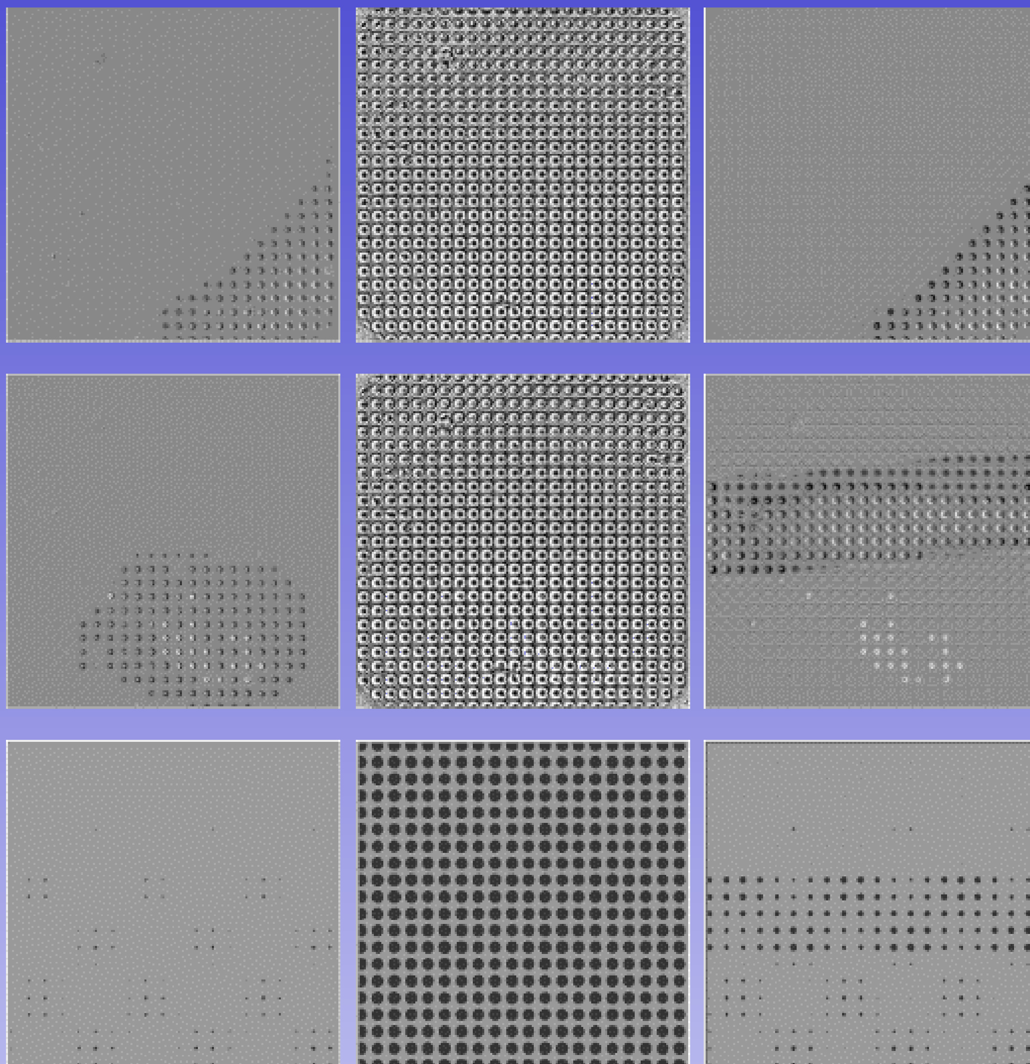
Conditions:

dd/dt = 1 mm/s

$h_{PS\ strip}$ = 30 nm

Temperature ~ 80°C

Qualitative Analysis

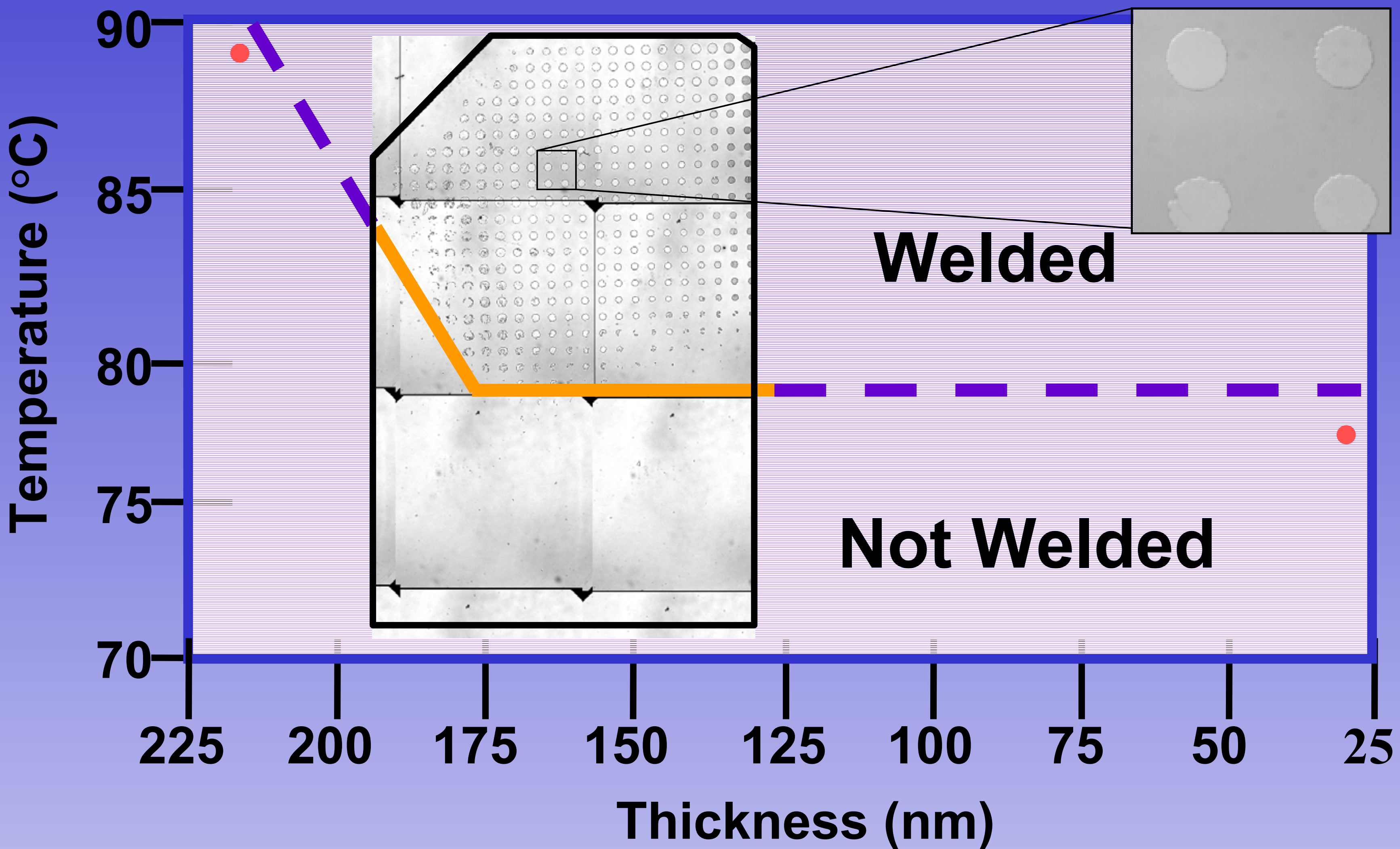


(a) Room Temperature

(b) Elevated Temperature

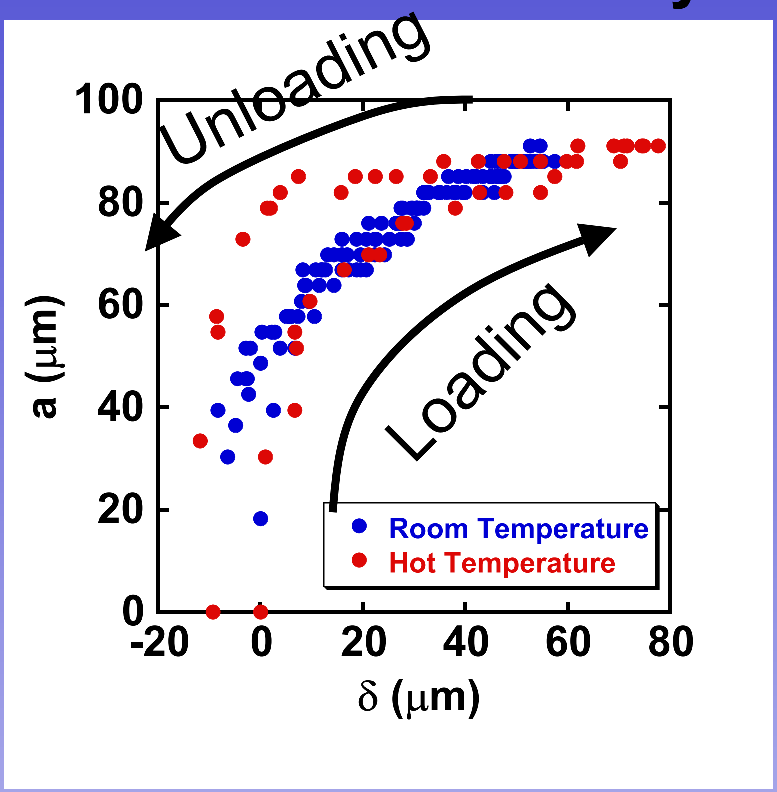
(c) Simulation

Mapping Multivariable Environments

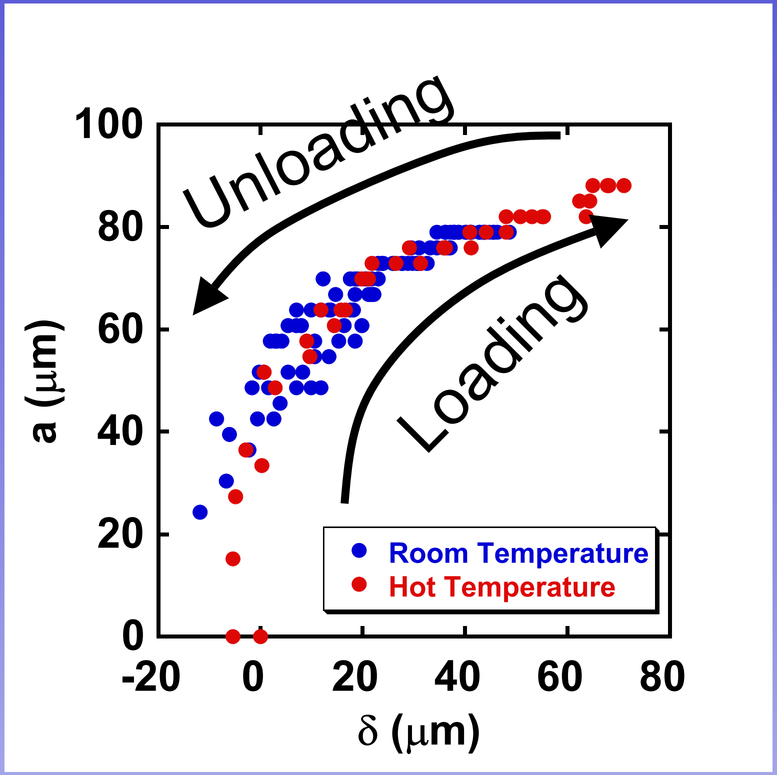


Quantitative Measurements

PS/PS Contact History

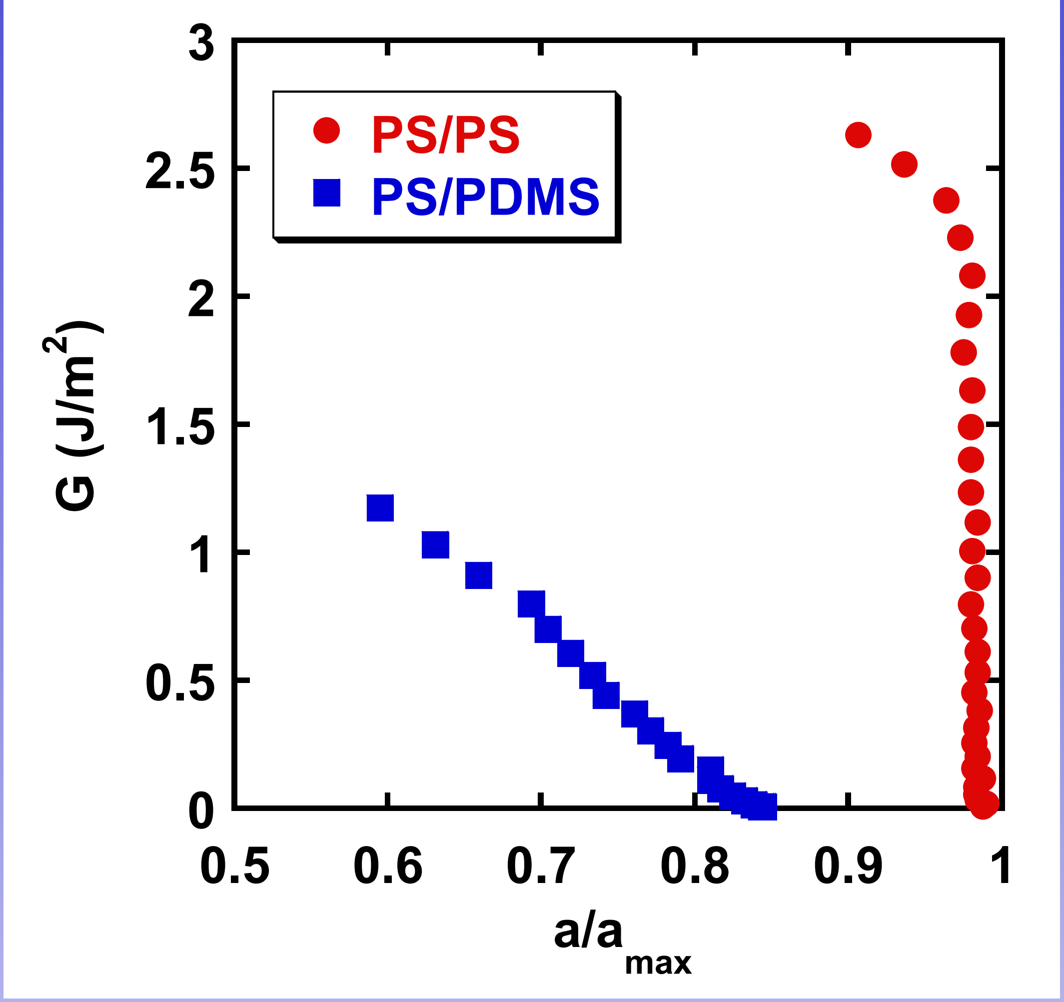


PS/PDMS Contact History



Same Sample, Same Conditions!

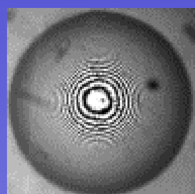
Quantitative Measurements

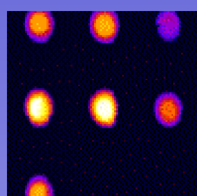


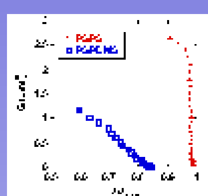
All weld spots fracture at same applied energy release rate.

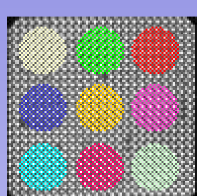
Time-Temperature dependence for interfacial strength development indicated by changing weld spot size and thickness dependence.

Outline









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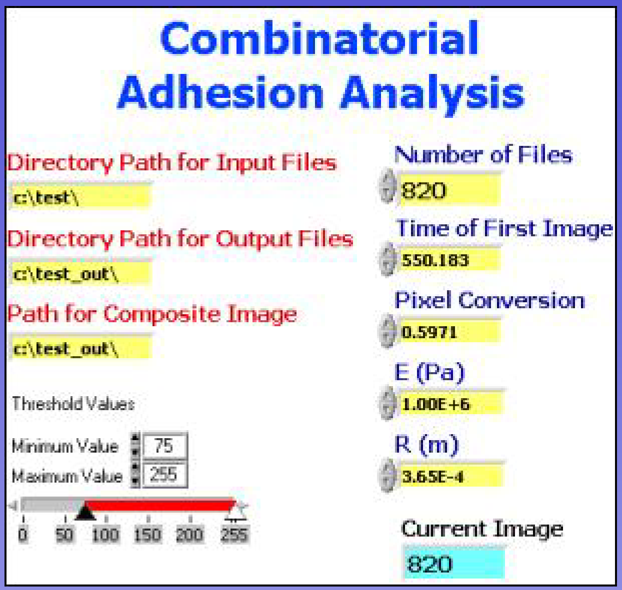
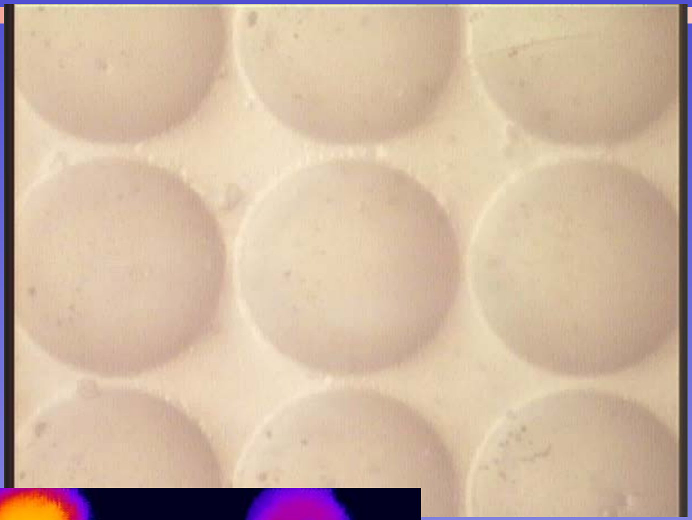
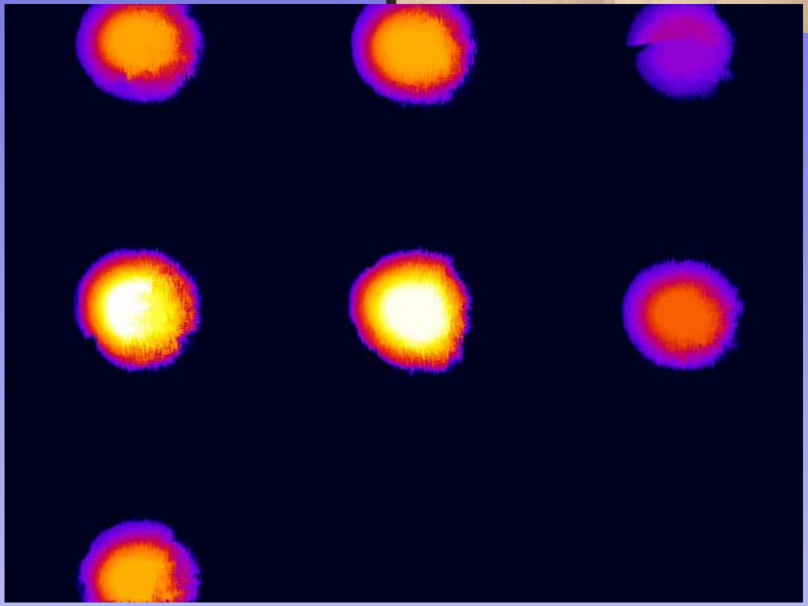
Automated Analysis





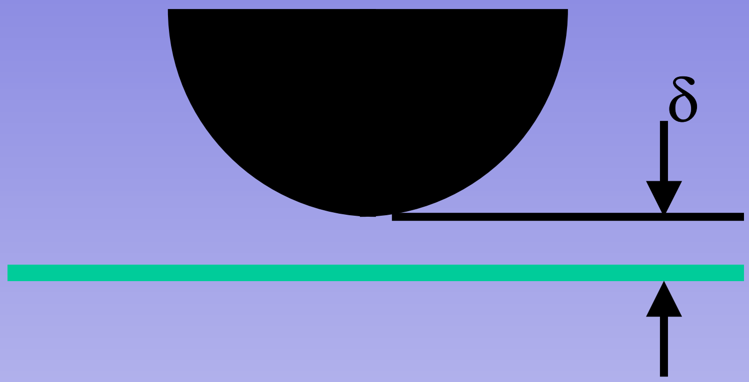


"Jump" Into Contact

JKR "Zero Force" contact radius

$$a_o = \left(\frac{27\pi R^2 \mathcal{G}}{8E}\right)^{1/3}$$

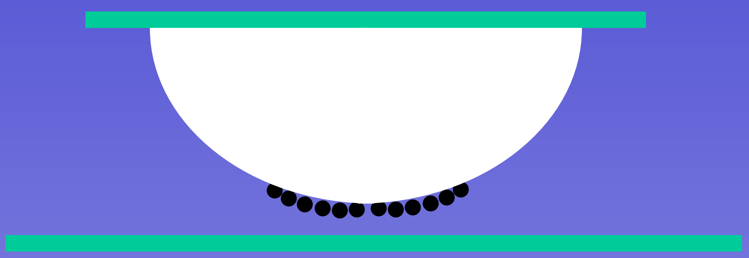
For soft, elastic solids, G is defined by thermodynamic work of adhesion.



$\delta_{critical} = \delta$ at "jump" into contact

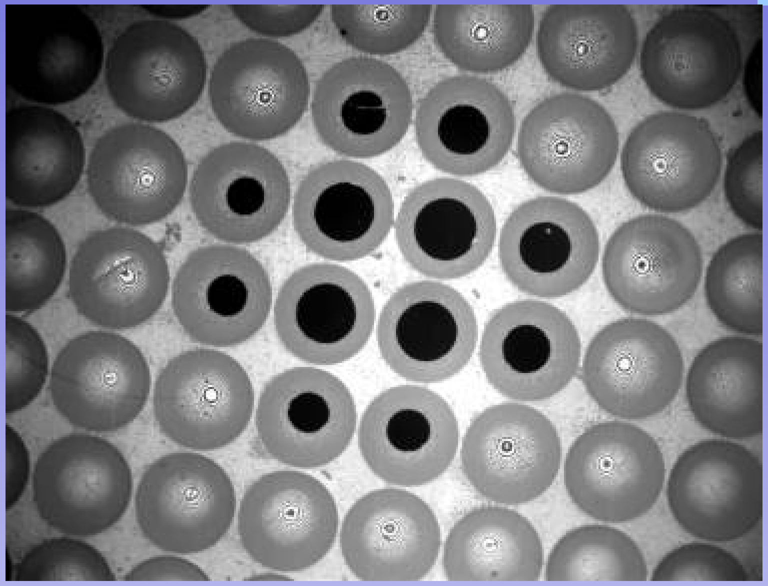
Defined by balance of surface and elastic restoring forces

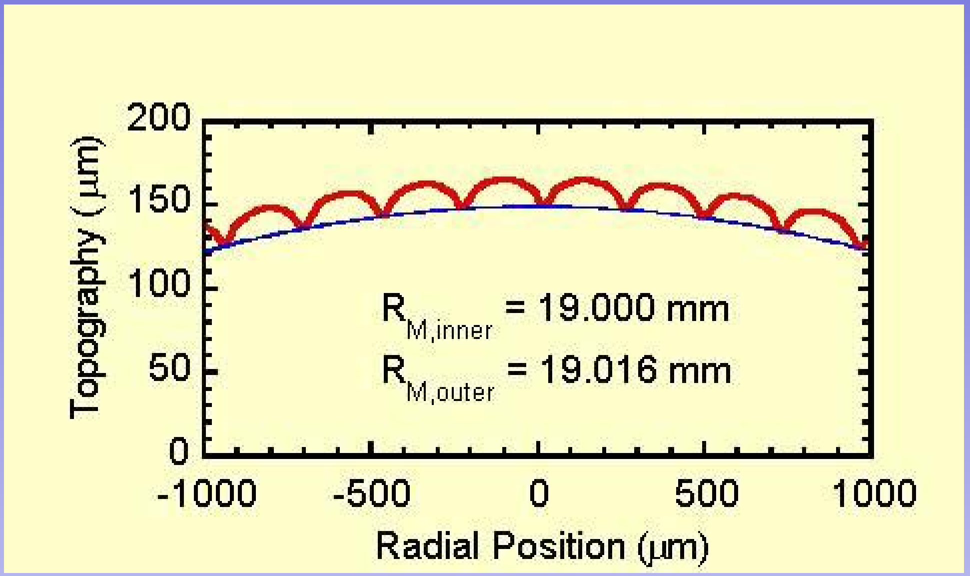
MultiLens Contact Technique



Advantages of Multilens Contact

- Maximize sensitivity and visualize contact
- Investigate effects of roughness on surface interactions
- Investigate dynamics of surface attraction and separation





Materials

Lens Arrays

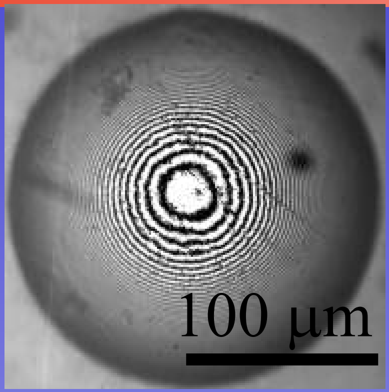
- Crosslinked Polydimethylsiloxane (PDMS)
- E = 1.0 Mpa
- $\gamma \sim 20$ mJ/m^2

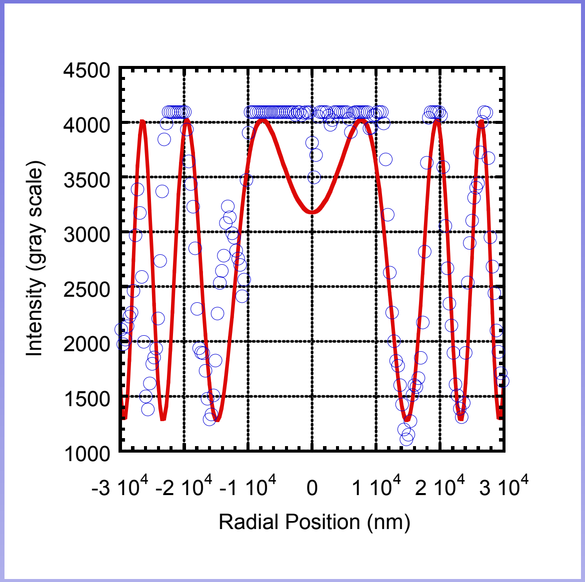
Substrates

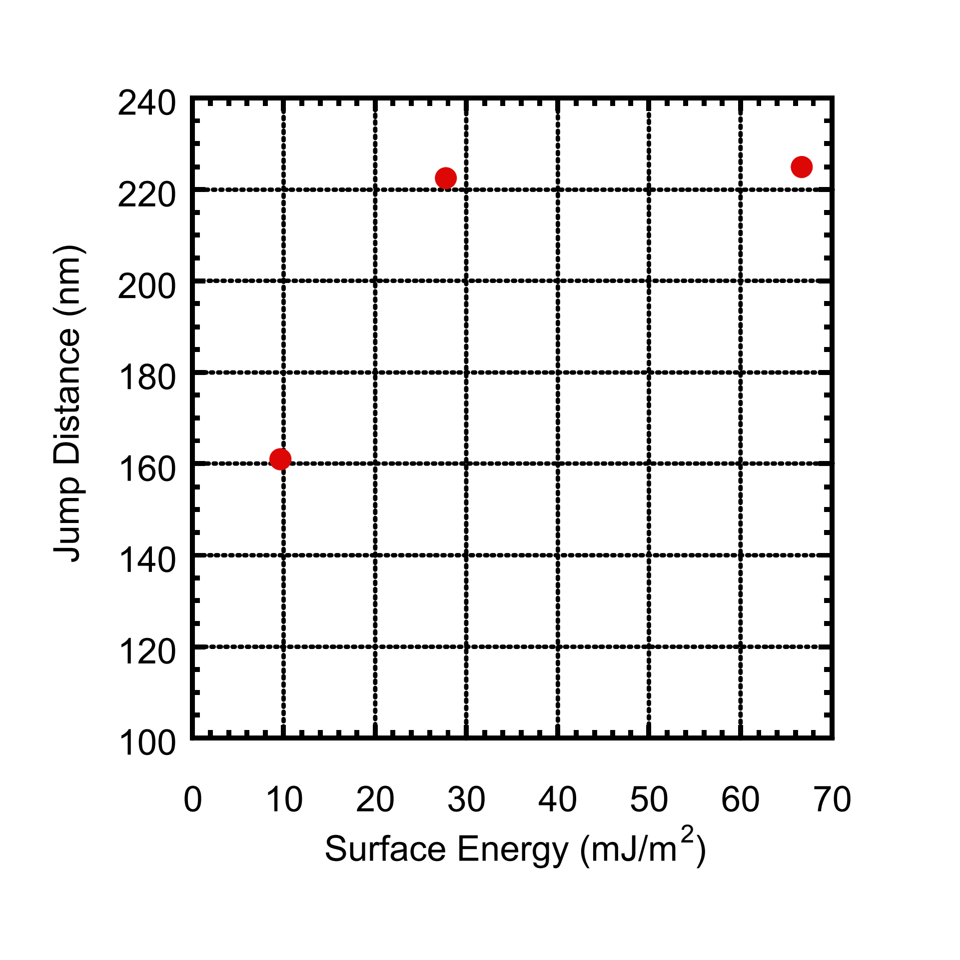
- Bare glass slide (ozone cleaned)
  - $\gamma = 67$ mJ/m^2
- n-octyl dimethylchlorosilane coated glass slide
  - $\gamma = 28$ mJ/m^2
- Fluorinated glass slide
  - $\gamma = 10$ mJ/m^2

Quantifying Jump Distance

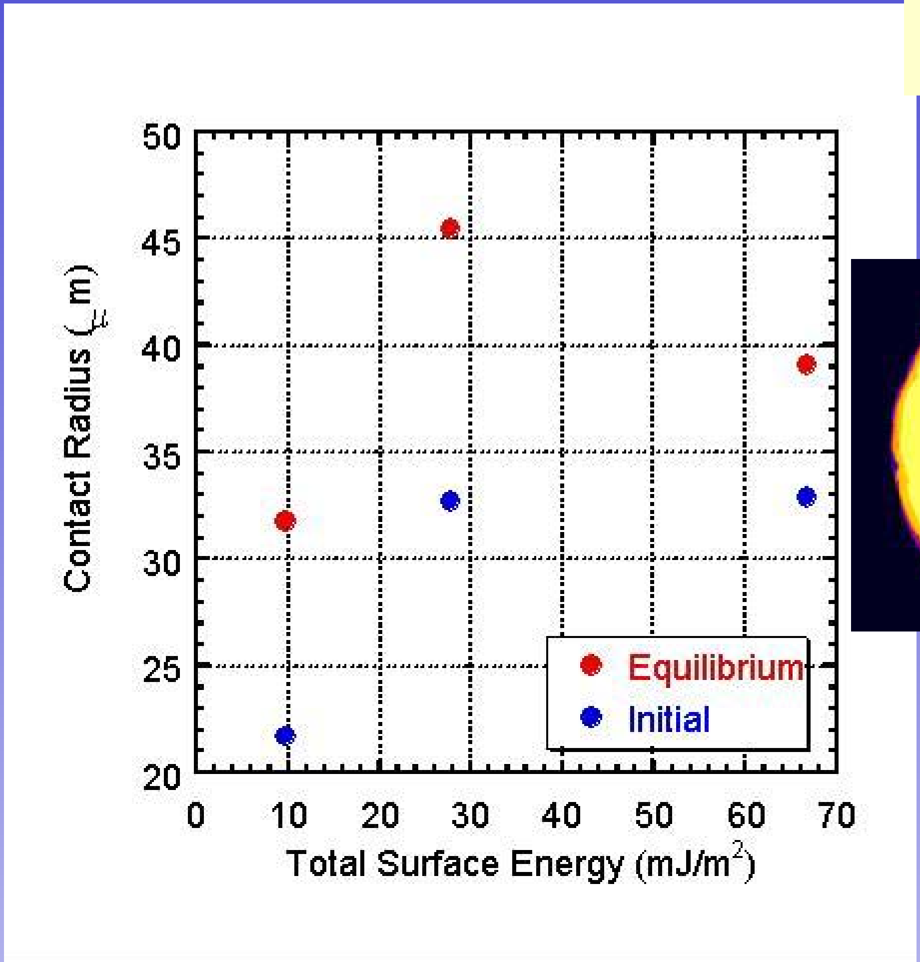
Approach substrate at 4 nm/s

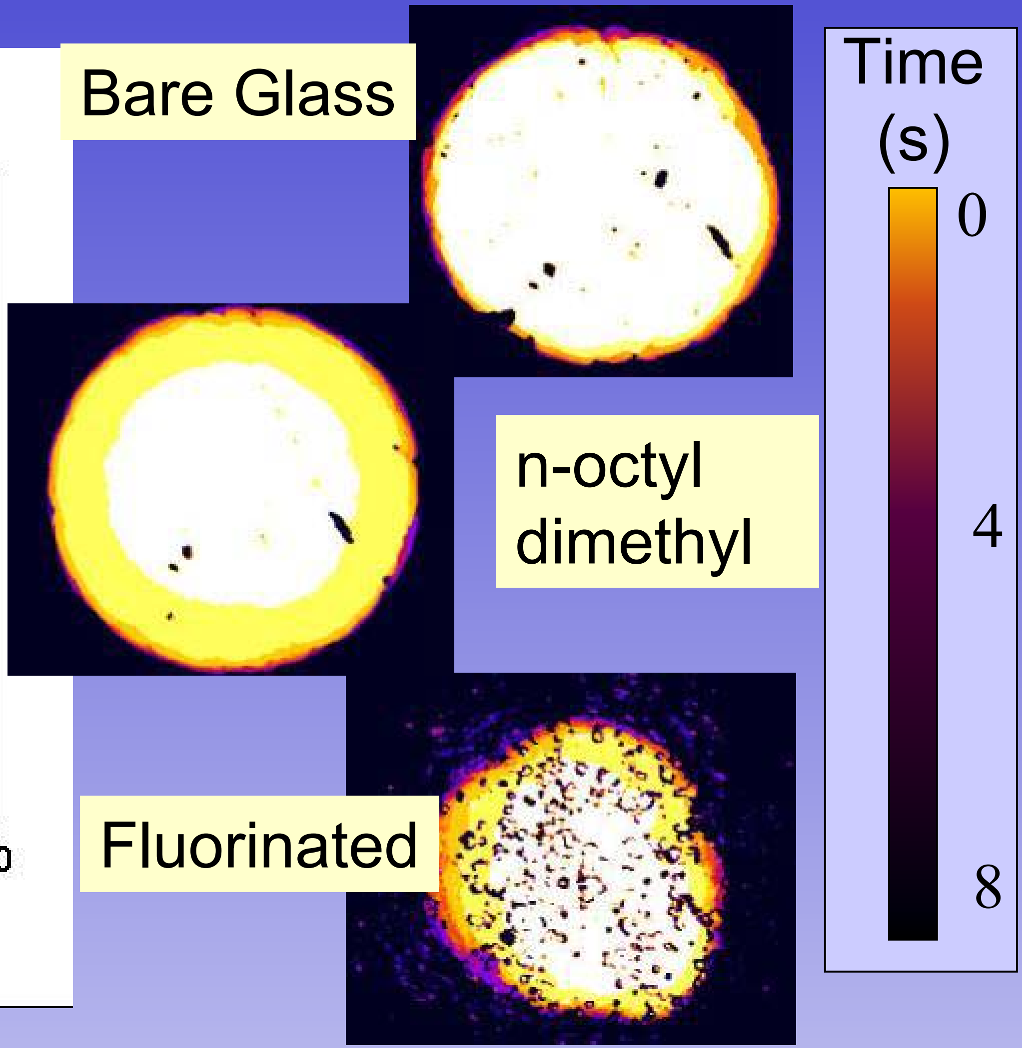




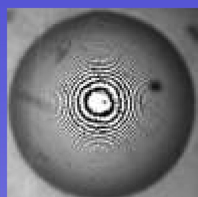


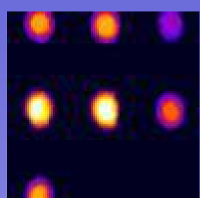
Dynamics of Contact

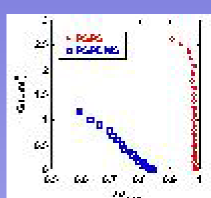


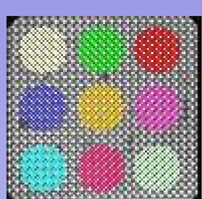


Outline



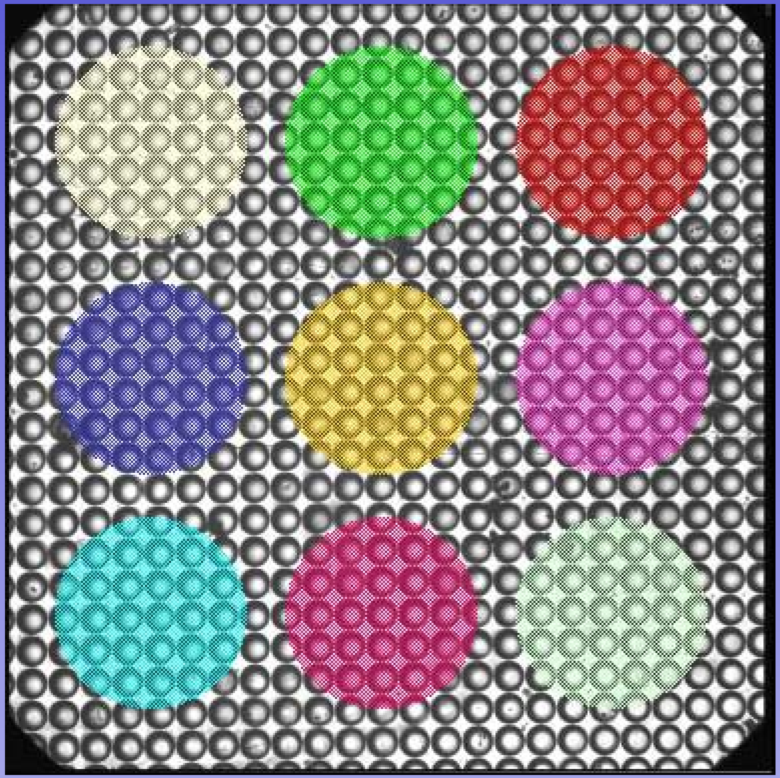






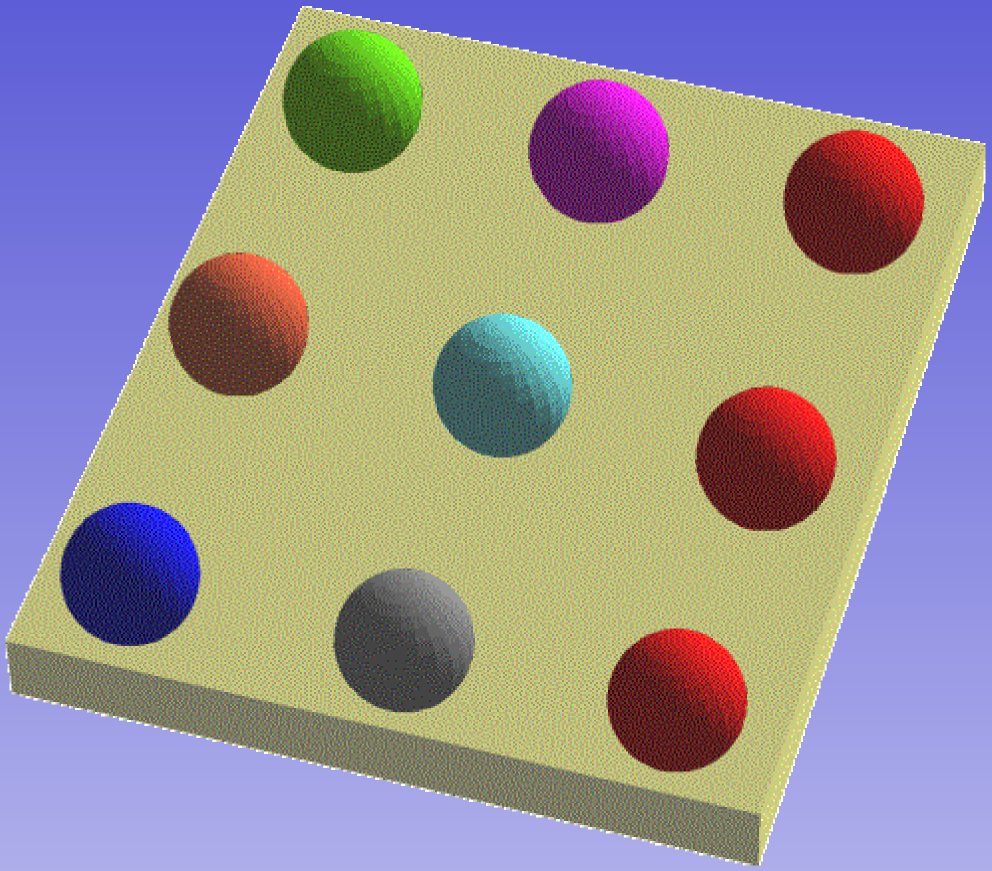
- Conventional Adhesion Tests
- Multilens Contact Adhesion Test Theory and Method
- Examples and Implementation
- High-Throughput Analysis
- Practical Points
- Summary

Practical Points



- Every point does not need to be different.
- Onset of cavitation or fingering is related to strain/stress of "tack force"
- Contact adhesion tests can be used for both weak and strong adhesion
- Control $\mathcal{G}$ to measure mechanical properties

Practical Points



- Size of lens and array can be modified
- A row of standard contacts can be incorporated
- Either lenses or substrate can be designed with softer materials

Summary

- Quantitative and qualitative adhesion testing is difficult
- Axisymmetric adhesion tests offer enhanced standardization and information
- Combinatorial approaches can simplify screening processes
- MCAT methodology is general